VOLATILE ORGANIC COMPOUND ANALYSES - U.S. EPA SW-846, Method 8260B.

Volatile organic compound (VOC) analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOP is equivalent to and referenced as EPA SW-846 Method 8260B for analysis of purgeable organic compounds.

Three catch basin sediments were analyzed for volatile organics, which includes two primary sample locations and one blind duplicate. Sample results are presented with associated data qualifiers in Table 4-14.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and complete. Maximum holding times are specified as 14 days at 4° C. (±2° C.) for solids. Upon receipt at the laboratory, transport cooler temperatures ranged from 4 to 6.2 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

GC/MS Tuning: GC/MS tune performance was checked with Bromofluorobenzene (BFB) prior to analysis of project samples. All sample analyses were performed within 12 hours of BFB analyses. All ion abundances and relative ion abundances meet method requirements. Review of mass spectral plots and associated mass listings supplied with the raw data shows no inconsistencies or errors. (Note: Instrument I.D. is not presented on documentation. It is assumed that the same instrument was employed for these analyses.)

Initial Calibration: The laboratory performed initial multipoint calibration at 0.4, 0.8, 2.0, 5.0, 10, and 20 µg/kg for all target analytes. Surrogate compounds were only analyzed at 5.0 µg/kg in each calibration run. Average Relative Response Factors (Average RRFs) are specified to be =0.05, and Relative Standard Deviations (RSDs) must be =15% for volatile Target Compound List (TCL) analytes. Average RRFs are =0.05 for all TCL analytes in the initial calibration (performed on May 11, 2001; just prior to sample analyses) with the exception of acetonitrile and 2-butanone (Average RRF = 0.015 & 0.024, respectively). Acetonitrile is not a project target analyte, and 2-butanone was reported in all samples. Analytes with associated positive results and with RSDs > 15% include 2-butanone, acetone, and bromomethane (RSD = 25%, 52% & 18%, respectively). Associated results are consequently qualified as estimates with a "J" qualifier code.

A 0.2 µg/kg linearity verification check was run for all target analytes prior to sample analyses. All target analytes exhibited acceptable response, with the exception of the ketones, which requires qualification of all ketone results less than 0.4 µg/kg as estimated ("J" qualifier code).

Continuing Calibration: Continuing calibration checks were performed prior to and following sample analyses (@ 2.0 µg/kg for target analytes and 5.0 µg/kg for surrogates). Project specifications are RRF must be =0.05, and Percent Differences (%Ds) must be $\leq$25% for volatile TCL analytes. RRFs are =0.05

for all compounds, with the exception of those analytes previously identified as deviant in the initial calibration. For analytes with associated positive results, %Ds are $\leq$25% in all continuing calibrations with the exception of acetone. Accordingly, all acetone results require qualification as estimates ("J" qualifier code).

Blanks: An analytical method blank was run prior to sample analyses. Detected analytes and associated concentrations are as follows:

bromomethane	0.6 µg/kg
iodomethane	0.8 µg/kg
vinyl acetate	450 µg/kg
2-butanone	4.3 µg/kg
toluene	0.2 µg/kg
bromoform	0.6 µg/kg

Method blank results are evaluated relative to project samples with associated positive hits. Positive results for bromomethane in 010504SBDS24SS, 010501SBMS01SS and 010501SBMS02SS, and 2-butanone in 010501SBMS01SS and 010501SBMS02SS are qualified as nondetects ("U") due to method blank performance.

No field or transport blanks were submitted from the field.

Surrogate Compound Performance: Surrogate compounds were added to each sample prior to analysis to assess analytical performance on each sample. The surrogate compounds d_8-toluene, bromofluorobenzene, d_{10}-ethylbenzene, fluorobenzene, and dibromofluoromethane have the following acceptable recovery ranges for solids: d_8-T (91-109%), BFB (80-113%), d_{10}-EB (0-106%), FB (85-115%), and DBFM (75-115%). Surrogate performances were within acceptable ranges, with the exception of a slightly high recovery (119%) in 010501SBMS02SS. No results require qualification due to surrogate performance.

Matrix Spike/Matrix Spike Duplicate Analyses: One MS/MSD pair was analyzed for the sample delivery group, as specified. All TCL compounds were added to the samples, however only selected analytes were evaluated for determination of performance. MS/MSD performance is evaluated relative to the specifications of the USACE Shell for Analytical Chemistry. Control limits applied are from the Shell (70-130% recovery). Recoveries are all within the acceptable ranges, with the exception of toluene, which was not able to be evaluated due to a native level at a significantly higher concentration (24 µg/kg) than the spike level (2.7 µg/kg). MS/MSD performance is considered acceptable.

Laboratory Control Samples: A spiked blank (LCS) and LCS duplicate were analyzed with the sample delivery group. Recoveries for the same analytes evaluated in the MS/MSD analyses showed 89 - 113% for both LCS's at a spike level of 2 µg/kg. LCS performance is considered acceptable and the analytical systems are determined to be in control.

TCL Compound Identification: The relative retention times (RRTs) for all reported TCL compounds are within acceptance limits (±0.06 RRT units), and were all within 2 seconds of the expected retention

times. All mass spectra show good comparison with library reference spectra. Ion relative abundances on mass spectra for all reported compounds compare acceptably to library reference spectra. It is noted that the analyst failed to identify bromomethane in the method blank and yet correctly identified and reported it in the site samples. Bromomethane in the site samples are associated with lab background contamination and are consequently qualified as nondetects at the associated values ("U" qualifier code - see method blank, above).

Compound Quantitation and Reported Detection / Quantitation Limits: Reported quantitation or lower reporting limits are determined to be actual lower reporting limits with associated verifiable linear calibration points (no extrapolations observed). All reported concentrations less than the verifiable linear calibration range are appropriately qualified by the lab with the "J" code.

System Performance: Raw data show no indication of degradation of system performance during or between analytical runs. Reconstructed ion chromatograms (RICs) show no abrupt shifts in baseline, high background levels, excessive baseline rise with increased temperature, or other indications of system performance degradation.

A comparison of results from the analyses of split samples of 010501SBMS01SS by the project lab and a reference laboratory are summarized below:

Analyte	Project lab	Ref. lab
Dichlorodifluoromethane	0.51 U	10 U
Chloromethane	0.51 U	5.2 U
Vinyl chloride	0.51 U	5.2 U
Bromomethane	0.86 U	5.2 U
Chloroethane	0.51 U	10 U
Trichlorofluoromethane	0.51 U	5.2 U
Acetone	**59 J**	**22 J**
1,1-Dichloroethene	0.51 U	5.2 U
Methylene chloride	0.51 U	5.2 U
Carbon disulfide	**1.5**	5.2 U
trans-1,2-Dichloroethene	0.51 U	5.2 U
Vinyl acetate	2.5 U	52 U
1,1-Dichloroethane	0.51 U	5.2 U
2-Butanone	10 U	52 U
2,2-Dichloropropane	0.51 U	5.2 U
cis-1,2-Dichloroethene	0.51 U	5.2 U
Chloroform	0.51 U	5.2 U
Bromochloromethane	0.51 U	5.2 U
1,1,1-Trichloroethane	0.51 U	5.2 U
1,1-Dichloropropene	0.51 U	5.2 U
Carbon tetrachloride	0.51 U	5.2 U
1,2-Dichloroethane	0.51 U	5.2 U
Benzene	**0.94**	5.2 U
Trichloroethene	0.51 U	5.2 U

1,2-Dichloropropane	0.51 U	5.2 U
Bromodichloromethane	0.51 U	5.2 U
Dibromomethane	0.51 U	5.2 U
4-Methyl-2-pentanone	2.5 U	52 U
cis-1,3-Dichloropropene	0.51 U	5.2 U
Toluene	**24**	**17.1**
trans-1,3-Dichloropropene	0.51 U	5.2 U
2-Hexanone	2.5 U	52 U
1,1,2-Trichloroethane	0.51 U	5.2 U
1,3-Dichloropropane	0.51 U	5.2 U
Tetrachloroethene	**0.31 J**	5.2 U
Dibromochloromethane	0.51 U	5.2 U
1,2-Dibromoethane	0.51 U	5.2 U
Chlorobenzene	0.51 U	5.2 U
Ethylbenzene	**0.41 J**	5.2 U
1,1,1,2-Tetrachloroethane	0.51 U	5.2 U
meta-/para-Xylenes	**0.56 J**	5.2 U
ortho-Xylene	**0.30 J**	5.2 U
Styrene	0.51 U	5.2 U
Isopropylbenzene	0.51 U	5.2 U
Bromoform	0.51 U	5.2 U
1,1,2,2-Tetrachloroethane	0.51 U	5.2 U
1,2,3-Trichloropropane	0.51 U	5.2 U
n-Propylbenzene	0.51 U	5.2 U
Bromobenzene	0.51 U	5.2 U
1,3,5-Trimethylbenzene	**0.29 J**	5.2 U
2-Chlorotoluene	0.51 U	5.2 U
4-Chlorotoluene	0.51 U	5.2 U
tert-Butylbenzene	0.51 U	5.2 U
1,2,4-Trimethylbenzene	**0.40 J**	5.2 U
sec-Butylbenzene	0.51 U	5.2 U
p-Isopropyltoluene	**9.5**	**3.0 J**
1,3-Dichlorobenzene	0.51 U	5.2 U
1,4-Dichlorobenzene	0.51 U	5.2 U
n-Butylbenzene	0.51 U	5.2 U
1,2-Dichlorobenzene	0.51 U	5.2 U
1,2-Dibromo-3-chloropropane	0.51 U	26 U
1,2,4-Trichlorobenzene	0.51 U	26 U
Hexachlorobutadiene	0.51 U	26 U
Naphthalene	**0.70**	26 U
1,2,3-Trichlorobenzene	0.51 U	26 U

Results compare reasonably well, especially for a solid. Only two analytes exhibited detections above both labs' lower quantitation limits. Lower reporting limits varied by a factor of ten between the two labs.

Field Replicates: A blind field replicate sample pair was submitted and analyzed for VOCs for determination of analytical variability. Results for the pair are determined to be relatively comparable, with the exception of acetone and toluene, which showed 74% and 85% differences, respectively (59 / 27 µg/kg for acetone and 24 / 9.5 µg/kg for toluene). These deviations are not atypical of the variabilities observed for contaminated solids.

Overall Assessment: All deliverables required by the project are present and data packages are complete. Recommended sample holding times and conditions were met. GC/MS tuning requirements were met. Initial and continuing calibration performances are acceptable with some qualification of data. Method blank analysis showed some background contamination for detected target analytes. As a result, several associated sample results required qualification as not detected ("U"). Overall, surrogate compound recoveries are acceptable. MS/MSD and LCS performances were acceptable. Compound identification and quantitation are acceptable. Raw data show no indications of system performance degradation. Reported quantitation or lower reporting limits are verifiable and relatively low for these types of analyses. Replicate analysis was performed on one sample pair and showed typical performance for contaminated solids. Overall analytical performance is considered acceptable, and data quality is sufficient for project use.

A summary of qualified results is as follows:

Sample	Analyte	Value	Qualifier	Deviation
010501SBMS01SS	Bromomethane	0.86	U	Blank contamination
	Acetone	59	J	Initial calibration
	2-Butanone	10	J	Initial calibration
	Tetrachloroethene	0.31	J	< PQL
	Ethylbenzene	0.41	J	< PQL
	m,p-Xylene	0.56	J	< PQL
	o-Xylene	0.30	J	< PQL
	1,3,5-Trimethylbenzene	0.29	J	< PQL
	1,2,4-Trimethylbenzene	0.40	J	< PQL
010501SBMS02SS	Bromomethane	0.76	U	Blank contamination
	Acetone	27	J	Initial calibration
	2-Butanone	7.5	J	Initial calibration
	Ethylbenzene	0.4	J	< PQL
	m,p-Xylene	0.52	J	< PQL
	o-Xylene	0.26	J	< PQL
	Naphthalene	0.27	J	< PQL
010504SBDS24SS	Bromomethane	1.8	U	Blank contamination
	Acetone	130	J	Initial calibration
	2-Butanone	25	J	Initial calibration
	Benzene	0.58	J	< PQL

PETROLEUM HYDROCARBON ANALYSES – WADOE NWTPH-HCID & NWTPH-Dx.

Petroleum hydrocarbon analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOPs are equivalent to and referenced as WADOE Northwest TPH-HCID and TPH-Dx (NWTPH-Dx [diesel range = C_{12} - C_{24} (as #2 diesel), lube or motor oil range = C_{24} - C_{38}]) - Semivolatile Petroleum Products Method for Soil and Water Analyses [with sulfuric acid and silica gel cleanup], as established by the Washington State Department of Ecology. NWTPH-HCID analyses were applied for screening with confirmatory results provided by NWTPH-Dx. NWTPH-HCID results indicated presence of diesel-range and lube-range hydrocarbons, with no gasoline range hydrocarbons greater than the lower reporting limits. This evaluation is performed for NWTPH-Dx only. No NWTPH-HCID evaluation is performed here. All NWTPH-Dx chromatograms were evaluated for determination of presence of specific or identifiable hydrocarbon mixtures. All bolded/highlighted values indicate the presence of the specific hydrocarbon mixture reported. Non-highlighted values represent presence of organics in the respective analytical range, but presence of a petroleum hydrocarbon mixture is determined to be improbable.

Five catch basin sediments were analyzed for diesel fuel and motor or lube oil hydrocarbons, which includes four primary sample locations and one blind duplicate. Sample results are presented with associated data qualifiers in Table 4-16.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and generally complete. Maximum holding times for petroleum hydrocarbons are specified as 14 /40 days (sample / extract maximum holding times) for solids at 4 °C. (±2 °C.). Upon receipt at the laboratory, transport cooler temperatures ranged from 4 to 6.2 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

Initial Calibration: The laboratory performed initial multipoint calibration (linearity verification) for #2 diesel fuel at 25, 50, 100, 250, 500, 1000, 2500 and 5000 µg/mL; and motor oil at 50, 100, 250, 500, 1000, 2500 and 5000 µg/mL (both on 4/20/02). Linearity for diesel was 8.1 RSD, and r^2 = 1.00 for motor oil. Initial calibration levels, frequencies, and linearities are within pertinent guidance limits.

Calibration Checks: Calibration verifications were performed at concentrations of 1000 µg/mL for diesel and motor oil prior to and following sample runs, and at a frequency of every ten analyses. Criteria for passing are $\pm$ 15% from the initial calibration. %Diff were all $\leq$ 7%. No results required qualification based on out-of-compliance procedures and performance criteria.

Blanks: Two analytical method blanks were analyzed, as required. No analyte responses were reported for method blanks.

No field rinsate or transport blanks were submitted nor analyzed.

Surrogate Compound Performance: Surrogate compounds were added to each sample prior to analysis to assess analytical performance on each sample. The surrogate compounds for petroleum hydrocarbon analyses are 1-chlorooctane and o-terphenyl for diesel and motor oil. Surrogate performance is evaluated for o-terphenyl only with an acceptance range of 50-150% recovery. All recoveries are within specification.

Matrix Spike/Matrix Spike Duplicate Analyses: Three MS/MSD pairs were analyzed. Diesel and motor oil were added to selected samples for evaluation of MS/MSD performance at 620 -1260 mg/kg. Control limits applied are 50-150% recovery with a %D of less than 50%. Recoveries are all within the acceptance range. MS/MSD performance is considered acceptable.

Laboratory Control Samples: Two spiked blanks (LCSs) were analyzed. Both LCSs showed recoveries in the range of 95 - 119% for both diesel and motor oil. LCS spiking levels were 500 mg/kg. Performance is considered acceptable and the analytical systems are determined to be in control.

Petroleum Hydrocarbon Mixture Identification: Positive identifications of hydrocarbon mixtures are highlighted by applying bold-face type to the values reported in the attached table. No diesel hydrocarbons are identified, however four of the five samples showed presence of a lube-range mixture that could include hydraulic, dielectric and/or pump fluids.

System Performance: Raw data show no indication of degradation of system performance during or between analytical runs. Chromatograms show no abrupt shifts in baseline, high background levels, excessive baseline rise with increased temperature, or other indications of system performance degradation.

A comparison of results from the analyses of split samples by the project lab and an independent reference laboratory shows the following (mg/kg, dry):

	010501SBMS01SS		010503IW14SS	
	Project Lab	Ref. Lab	Project Lab	Ref. Lab
Diesel-range	130	100	61 U	10
Lube-range	**600**	**230**	120 U	50 U

Both labs identified lube oil in 01SS, and no recognizable petroleum product in 14SS.

Field Replicates: One blind field replicate sample pair was submitted and analyzed for petroleum hydrocarbons for determination of analytical variability. The duplicate pair showed comparable results.

Overall Assessment: All deliverables required by the project are present and data packages are complete. Recommended sample holding times and conditions were met. Initial calibration and calibration check requirements were met. Method blank performances were within specification. Surrogate compound recoveries are acceptable. MS/MSD and LCS performances are acceptable. Compound identification and quantitation are acceptable. Raw data show no indications of system performance degradation. Overall analytical performance is considered acceptable, and data quality is sufficient for project use.

SEMIVOLATILE ORGANICS ANALYSES - U.S. EPA SW-846, Method 8270C.

Semivolatile organics analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOP is equivalent to and referenced as EPA SW-846 Method 8270C for analysis of acid, base and neutral extractable organic compounds. Extract preparations were performed in accordance with SW-846 Method 3550B.

Five river and five catch basin sediments were analyzed for semivolatile organics, which includes eight primary sample locations and two blind duplicates. Sample results are presented with associated data qualifiers in Tables 4-4 and 4-13.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and generally complete, with the exception of two missing initial calibration runs. A request to the lab for submission of missing documentation was made to complete the data package. Maximum holding times for extractables are specified as 14 /40 days (sample / extract maximum holding times) for solids at 4 °C. (±2 °C.). Upon receipt at the laboratory, transport cooler temperatures ranged from 4 to 6.2 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

GC/MS Tuning: GC/MS tune performance was checked with 2.5 ng decafluorotriphenylphosphine (DFTPP) prior to all initial calibrations runs and all subsequent sample analytical runs. All sample analyses were performed within 12 hours of DFTPP checks. All ion abundances and relative ion abundances meet method requirements. Review of mass spectral plots and associated mass listings supplied with the raw data show no inconsistencies or errors.

Initial Calibration: Initial multipoint calibrations were performed at 0.05, 0.1, 0.5, 1.0 and 2.5 µg/mL [on 5/3/01] and 0.1, 0.25, 0.5, 1.0, and 2.5 µg/mL [on 5/10/01] for all target analytes and surrogate compounds. Average Relative Response Factors (Average RRFs) must be =0.01, and selected analytes must meet additional minimum RRF and maximum %RSD criteria. Average RRFs and %RSDs for all TCL compounds in all initial calibrations showed compliance with technical requirements.

Continuing Calibration: Continuing calibrations were performed for all TCL compounds at 1.0 ng/µL. RRFs must be =0.01, and selected analytes must meet additional minimum RRF and maximum %Diff criteria ($\leq$25%). All RRFs were in compliance with some deviation from criteria for %Diff. Noncompliant %Diff values did not affect sample results, since no positive detects were reported for the affected analytes.

Blanks: Analytical method blanks were analyzed at least once for each analytical group and matrix, as specified. Method blanks showed some detections of phthalates (butylbenzyl- & *bis*(2-

ethylhexyl)phthalate) above reporting limits. Reporting limits were adjusted upward for phthalates to minimize potential bias associated with background contamination.

Surrogate Compound Performance: Surrogate compounds (1.0 µg each or ~67 µg/kg, wet) were added to each solid sample prior to extraction to assess analytical performance on each sample. Surrogate compounds and associated performance criteria are those specified in the ACOE Shell for Analytical Chemistry (for solids). No results required qualification due to surrogate compound recovery performance. (It is noted that surrogate recoveries tended to be higher than normally observed for these types of analyses, and were generally greater than 100%. Recoveries ranged from 77% to 214% with a median greater than 100%).

Matrix Spike/Matrix Spike Duplicate Analyses: Matrix spike and matrix spike duplicate analyses were performed on three sediments. Analyte spike concentrations were 84, 91, and 170 µg/kg. MS/MSD recoveries were evaluated against the specifications in ACOE Shell for Analytical Chemistry. Recoveries ranged from 32% to 340%, even without the interference of native analytes in the extracts. Surrogate compound responses [recoveries] generally correlated with MS and MSD performances; typically greater than 100%. No results were qualified based on MS/MSD performance.

Laboratory Control Samples: Two spiked blanks (LCS) were analyzed for the solids analytical groups. LCS performance was compared to the specifications in the ACOE Shell for Analytical Chemistry. Spike concentrations were at an equivalent of 67 µg/kg. All recoveries were determined to be within acceptable range, generally averaged just less than 100%. LCS performance indicates the analytical systems are in control.

TCL Compound Identification: Relative retention times (RRTs) for most reported TCL compounds are acceptable (±0.06 RRT units or $\pm$ 6 seconds). Mass spectra, for some reported hits, show comparability with library reference spectra. Some target hits (potential positive identifications), such as 2,6-dinitrotoluene, N-nitrosodi-n-propylamine, and benzidine, were determined by the reviewer to be false positives. The 2,6-dinitrotoluene reports showed high interferences and noise across the baseline, a factor likely related to the use of an ITS40 (ion trap mass spectrometer) for analysis of "dirty" or busy extracts. The noise level/background is too high to determine presence at the reported values. The reported N-nitrosodi-n-propylamine is a poor mass spectral fit associated with an out-of-range retention time (+ 7 seconds relative to the calibration standard) and low signal to noise (S/N) response. The reports for benzidine are due to contributory ions (m/z 92 and 184) from the presence of a pentachlorobiphenyl isomer (a PCB constituent). Noise levels are sufficiently high for some PAHs to preclude the assignments made by the lab (for example, benzo(g,h,i)perylene in 010504SBDS24SS). The reported detection levels are generally lower than can be supported by the data. Spectral matches, in some cases, are marginal to poor. These values were replaced with appropriate nondetects ("U" qualified) for the analytes of concern. Benzo(b)fluoranthene and benzo(k)fluoranthene are insufficiently resolved chromatographically to report as distinguishable constituents. Caution should be exercised by the data user when interpreting results for these two analytes. A summation of results and use as a combined parameter (benzo(b+k)fluoranthenes) would be more appropriate.

Compound Quantitation and Reported Detection / Quantitation Limits: Lower reporting limits, in some cases, are lower than what can be verified by the reported data. The mass spectrometer used (an ion trap mass spectrometer - ITS40) is a very sensitive instrument prone to high background interferences when operated in an autogain mode. (The autogain function may have been engaged based on low S/N for some false positive assignments at significant reported concentrations [see N-nitrosodi-n-propylamine in 0105021IW14SS].) Essentially, the higher or more background the sample extract possesses (typical of sediments and soils), the more difficult it can be to identify presence of target analytes in the presence of interfering chemicals. Alternate instrumentations (such as quadrupolar, time of flight, or magnetic sector instruments) are better suited for analysis of complex extracts with high levels of interfering chemicals. Without documentation of mass spectrometer ionization times and autogain adjustment, detection limits may vary during an instrumental run without the knowledge of the analyst.

Lower reporting limits have been adjusted by the reviewer to concentrations equivalent to the lower limit of the initial calibrations (0.05 µg/mL). For some analytes (PAHs, in particular), the lab reported relatively low limits based on running a 0.01 µg/mL check standard. The 0.01 check standard is unverifiable in a complex sample extract. One-fifth of the surrogate concentrations (based on signal strength in extract runs) were determined to represent an average verifiable concentration for lower reporting limits for most analytes in each sample. These concentrations were also approximately equal to the equivalent level of the 0.05 µg/mL calibration standard. Consequently, lower reporting limits were adjusted by the reviewer to verifiable levels and not extrapolated or "clean extract" values.

Substituted anilines and phenols typically exhibit relatively low recoveries in environmental matrices. Most environmental analytical laboratories, including the reference laboratory, adjust their reported detection limits upward by factors of 10 or sometimes 20 for the difficult analytes relative to the "easy" analytes, such as PAHs. Practical quantitation levels (PQLs) should reflect the full sensitivity of the method and not extrapolated, or theoretical, limits. The project lab PQLs appear to be extrapolated and not verified PQLs. Note the differences in PQLs for the project lab compared to the reference laboratory for split samples below. Project lab PQLs should be considered unverified for analytes such as substituted phenols and anilines.

System Performance: The use of an ion trap mass spectrometer for the analysis of contaminated soils and sediments can result in the reporting of lower than actual quantitation/detection limits when the autogain function is engaged. This results in automatic, without necessarily the knowledge of the analyst, adjustment of instrumental sensitivities. This can result in variable and nonverifiable responses to target analytes (consistent with the results reported for MS/MSD and surrogate recovery performances) and higher than reported detection limits. The use of ion trap mass spectrometers, as they are currently available, is not recommended for analysis of contaminated solids without special extract cleanup procedures, which were not [reported to be] performed here. Consequently, the reported PQLs (practical quantitation limits) should be considered estimates, even those adjusted upward by the reviewer. The above recommendations and precautions are based on a review of reported mass spectral matches (and mismatches), false positive assignments, observation of high noise/background (low S/N) levels, and high surrogate and MS/MSD recovery variabilities.

A comparison of results from the analyses of split samples of 010501SBMS01SS and 010503IW14SS by the project lab and a reference laboratory are summarized below:

	010501SBMS01SS		010503IW14SS	
Analyte	**Project lab**	**Ref. lab**	**Project lab**	**Ref. lab**
Phenol	180 U	670 U	17 U	450 U
bis(2-Chloroethyl)ether	180 U	670 U	17 U	450 U
2-Chlorophenol	180 U	670 U	17 U	450 U
1,3-Dichlorobenzene	180 U	670 U	17 U	450 U
1,4-Dichlorobenzene	180 U	670 U	17 U	450 U
1,2-Dichlorobenzene	180 U	670 U	17 U	450 U
Benzyl alcohol	180 U	6700 U	17 U	4500 U
2-Methylphenol	180 U	670 U	17 U	450 U
2,2'-Oxy *bis*(1-chloropropane)	-	670 U	-	450 U
N-Nitrosodi-n-propylamine	180 U	670 U	42 U	450 U
Hexachloroethane	180 U	670 U	17 U	450 U
4-Methylphenol	**85 J**	**130 J**	17 U	450 U
Nitrobenzene	180 U	670 U	17 U	450 U
Isophorone	180 U	2000 U	17 U	1300 U
2-Nitrophenol	180 U	1300 U	17 U	890 U
2,4-Dimethylphenol	180 U	1300 U	17 U	890 U
bis(2-Chloroethoxy)methane	180 U	670 U	17 U	450 U
2,4-Dichlorophenol	180 U	670 U	17 U	450 U
Benzoic acid	450 U	6700 U	42 U	4500 U
1,2,4-Trichlorobenzene	180 U	670 U	17 U	450 U
Naphthalene	**27 J**	670 U	8 U	450 U
4-Chloroaniline	180 U	1300 U	17 U	890 U
Hexachlorobutadiene	180 U	670 U	17 U	450 U
4-Chloro-3-methylphenol	180 U	1300 U	17 U	890 U
2-Methylnaphthalene	180 U	670 U	8 U	450 U
Hexachlorocyclopentadiene	180 U	2700 U	17 U	1800 U
2,4,6-Trichlorophenol	180 U	670 U	17 U	450 U
2,4,5-Trichlorophenol	180 U	670 U	17 U	450 U
2-Chloronaphthalene	180 U	670 U	8 U	450 U
2-Nitroaniline	180 U	6700 U	17 U	4500 U
Acenaphthylene	**31 J**	670 U	8 U	450 U
Dimethylphthalate	180 U	670 U	17 U	450 U
2,6-Dinitrotoluene	180 U	670 U	17 U	450 U
3-Nitroaniline	180 U	6700 U	17 U	4500 U
Acenaphthene	**250**	**90 J**	8 U	450 U
2,4-Dinitrophenol	180 U	6700 U	17 U	4500 U
Dibenzofuran	**62 J**	670 U	17 U	450 U
4-Nitrophenol	180 U	6700 U	17 U	4500 U
2,4-Dinitrotoluene	180 U	670 U	17 U	450 U

Fluorene	**160**	670 U	8 U	450 U
Diethylphthalate	180 U	670 U	17 U	450 U
4-Chlorophenyl phenyl ether	180 U	670 U	17 U	450 U
4-Nitroaniline	180 U	6700 U	17 U	4500 U
4,6-Dinitro-2-methylphenol	450 U	6700 U	42 U	4500 U
N-Nitrosodiphenylamine	180 U	670 U	17 U	450 U
Hexachlorobenzene	180 U	670 U	17 U	450 U
4-Bromophenyl phenyl ether	180 U	670 U	17 U	450 U
Pentachlorophenol	180 U	6700 U	17 U	4500 U
Phenanthrene	**1100**	**470 J**	8 U	450 U
Anthracene	**290**	**90 J**	8 U	450 U
Di-n-butylphthalate	910 U	**120 JB**	84 U	**140 JB**
Fluoranthene	**2100**	**670**	8 U	450 U
Pyrene	**1900**	**750**	8 U	450 U
Butyl benzyl phthalate	180 U	670 U	17 U	450 U
Benzo(a)anthracene	**1000**	**330 J**	17 U	450 U
3,3'-Dichlorobenzidine	180 U	2700 U	17 U	1800 U
Chrysene	**1000**	**480 J**	8 U	450 U
bis(2-Ethylhexyl)phthalate	**3800**	**17200 B**	140 U	**220 JB**
Di-n-octylphthalate	180 U	**80 J**	17 U	450 U
Benzo(b)fluoranthene	**1700**	**690**	8 U	450 U
Benzo(k)fluoranthene	**370**	*(b+k)*	8 U	450 U
Benzo(a)pyrene	**1400**	**360 J**	8 U	450 U
Indeno(1,2,3-cd)pyrene	**640**	**240 J**	8 U	450 U
Dibenzo(a,h)anthracene	**180**	**80 J**	8 U	450 U
Benzo(g,h,i)perylene	**700**	**210 J**	8 U	450 U

Project lab results are generally greater than for the reference lab, which could be attributed to sample heterogeneity. The most notable difference between the two laboratory's data are the significantly lower detection limits reported by the project lab. Also, the project laboratory consistently reports lower reporting limits for "poor responders", such as substituted phenols and anilines, and other polars, at the same limits as for the higher responders, such as PAHs. The reference lab adjusts lower reporting limits for poor responders based on overall analytical system response, and not a theoretical or ideal limit.

Field Replicates: Blind field splits for two sediment pairs were submitted and analyzed for determination of analytical variability. Sample results for replicate pairs are presented in the attached results table. The sediment pairs showed typical variability for detected analytes in contaminated solids (variabilities up to 85% difference).

Overall Assessment: All deliverables required by the project are present and data packages are generally complete, with the exception of missing documentation for initial calibration runs performed on 5/10/01 that were later provided upon request, and mass spectrometer scan ionization times. Recommended sample holding times and conditions were met. GC/MS tuning requirements were met. Initial calibration requirements were generally met. Method blanks showed some low-level detections of

phthalates requiring the elevation of reporting limits for selected samples. Compound identification showed some false positives as noted above. Raw data shows some system performance degradation due to elevated noise levels, which interfered with the achievement of reported detection limits. Reported quantitation or lower reporting limits were adjusted upward for PAHs to verifiable levels. Lower reporting limits (detection limits) for many analytes are unverified; lower reporting limits for most of the polar analytes should be considered [gross] estimates and may be low by a factor of approximately 10. Overall analytical performance could be improved. The data as reported with associated qualifiers (following adjustments made by the reviewer) are adequate for project use.

A summary of qualified results is as follows:

Sample	Analyte	Value	Qualifier	Deviation
010501SBMS01SS	3- & 4-Methylphenol	85	J	< PQL
	Acenaphthylene	31	J	< PQL, nonverifiable
	2-Methylnaphthalene	180	U	PQL adjustment
	Dibenzofuran	62	J	< PQL
	2-Chloronaphthalene	180	U	PQL adjustment
	Di-n-butylphthalate	910	U	Blank contamination
	Benzidine	360	U	PQL adjustment
	Butylbenzylphthalate	180	U	Blank contamination
	Carbazole	190	J	< PQL
010501SBMS02SS	3- & 4-Methylphenol	69	J	< PQL
	Dibenzofuran	27	J	< PQL
	Fluorene	51	J	< PQL, nonverifiable
	2-Chloronaphthalene	170	U	PQL adjustment
	Acenaphthylene	170	U	PQL adjustment
	Butylbenzylphthalate	170	U	Blank contamination
	bis(2-Ethylhexyl)phthalate	2900	U	Blank contamination
	Carbazole	79	J	< PQL
010502IW01SS	2,6-Dinitrotoluene	16	U	False positive
	Diethylphthalate	16	U	Blank contamination
	2-Chloronaphthalene	8	U	PQL adjustment
	Acenaphthylene	8	U	PQL adjustment
	Acenaphthene	8	U	PQL adjustment
	Fluorene	8	U	PQL adjustment
	Anthracene	8	U	PQL adjustment
	Benzo(k)fluoranthene	8	U	PQL adjustment
	Di-n-butylphthalate	30	U	Blank contamination
	Butylbenzylphthalate	17	U	Blank contamination
	bis(2-Ethylhexyl)phthalate	42	U	Blank contamination
	Benzidine	16	U	False positive
010502IW07SS	3- & 4-Methylphenol	17	U	< PQL, nonverifiable
	2,6-Dinitrotoluene	17	U	False positive
	2-Chloronaphthalene	9	U	PQL adjustment
	Acenaphthylene	9	U	PQL adjustment

	Acenaphthene	9	U	PQL adjustment
	Fluorene	9	U	PQL adjustment
	Anthracene	9	U	PQL adjustment
	Benzo(k)fluoranthene	9	U	PQL adjustment
	Diethylphthalate	17	U	Blank contamination
	Di-n-butylphthalate	85	U	Blank contamination
	Butylbenzylphthalate	17	U	Blank contamination
	bis(2-Ethylhexyl)phthalate	190	U	Blank concentration
010503IW14SS	3- & 4-Methylphenol	17	U	< PQL, nonverifiable
	N-Nitrosodi-n-propylamine	42	U	False positive
	2,6-Dinitrotoluene	17	U	False positive
	2-Chloronaphthalene	8	U	PQL adjustment
	Acenaphthylene	8	U	PQL adjustment
	Acenaphthene	8	U	PQL adjustment
	Diethylphthalate	17	U	Blank contamination
	Fluorene	8	U	PQL adjustment
	Anthracene	8	U	PQL adjustment
	Di-n-butylphthalate	84	U	Blank contamination
	Fluoranthene	8	U	< PQL, nonverifiable
	Pyrene	8	U	< PQL, nonverifiable
	Butylbenzylphthalate	17	U	Blank contamination
	Chrysene	8	U	< PQL, nonverifiable
	bis(2-Ethylhexyl)phthalate	140	U	Blank contamination
	Benzo(k)fluoranthene	8	U	PQL adjustment
	Benzo(b)fluoranthene	8	U	< PQL, nonverifiable
010503IW15SS	3- & 4-Methylphenol	17	U	< PQL, nonverifiable
	2,6-Dinitrotoluene	17	U	False positive
	2-Chloronaphthalene	9	U	PQL adjustment
	Acenaphthylene	9	U	PQL adjustment
	Acenaphthene	9	U	PQL adjustment
	Diethylphthalate	17	U	Blank contamination
	Fluorene	9	U	PQL adjustment
	Phenanthrene	7	J	< PQL
	Anthracene	9	U	< PQL, nonverifiable
	Di-n-butylphthalate	87	U	Blank contamination
	Butylbenzylphthalate	17	U	Blank contamination
	Benzo(a)anthracene	7	J	< PQL
	Chrysene	7	J	< PQL
	bis(2-Ethylhexyl)phthalate	43	U	Blank contamination
	Benzo(k)fluoranthene	9	U	PQL adjustment
	Benzo(a)pyrene	5	J	< PQL
	Benzo(g,h,i)perylene	9	U	< PQL, nonverifiable
	Benzidine	70	U	False positive
010503IW16SS	2-Methylnaphthalene	8	U	< PQL, nonverifiable

	2-Chloronaphthalene	8	U	PQL adjustment
	Acenaphthylene	8	U	PQL adjustment
	2,6-Dinitrotoluene	16	U	False positive
	Acenaphthene	8	U	< PQL, nonverifiable
	Diethylphthalate	16	U	Blank contamination
	Fluorene	8	U	< PQL, nonverifiable
	Di-n-butylphthalate	78	U	Blank contamination
	Butylbenzylphthalate	20	U	Blank contamination
	bis(2-Ethylhexyl)phthalate	57	U	Blank contamination
	Benzo(b)fluoranthene	87		Sum of *b* & *k* isomers
	Benzo(k)fluoranthene			Unresolved from *b* isomer
010503SBDS18SS	2-Chloronaphthalene	170	U	PQL adjustment
	Acenaphthylene	170	U	PQL adjustment
	Acenaphthene	40	J	< PQL
	Dibenzofuran	83	U	< PQL, nonverifiable
	Fluorene	56	J	< PQL
	bis(2-Ethylhexyl)phthalate	2300	U	Blank contamination
	Benzo(b)fluoranthene	330		Sum of *b* & *k* isomers
	Benzo(k)fluoranthene			Unresolved from *b* isomer
	Indeno(1,2,3-cd)pyrene	80	J	< PQL
	Carbazole	71	J	< PQL
010503SBDS19SS	N-Nitrosodi-n-propylamine	40	U	False positive
	Naphthalene	6	J	< PQL
	2-Methylnaphthalene	9	U	< PQL, nonverifiable
	2-Chloronaphthalene	17	U	PQL adjustment
	2,6-Dinitrotoluene	17	U	False positive
	Dibenzofuran	9	J	< PQL
	Diethylphthalate	17	U	Blank contamination
	Di-n-butylphthalate	87	U	Blank contamination
	Butylbenzylphthalate	27	U	Blank contamination
	Carbazole	27	J	< PQL
010504SBDS24SS	2-Chloronaphthalene	340	U	PQL adjustment
	Acenaphthylene	170	U	PQL adjustment
	Acenaphthene	170	U	PQL adjustment
	Fluorene	170	U	PQL adjustment
	Phenanthrene	110	J	< PQL
	Benzo(a)anthracene	140	J	< PQL
	Benzo(k)fluoranthene	51	J	< PQL
	Benzo(a)pyrene	170	J	< PQL
	Indeno(1,2,3-cd)pyrene	34	J	< PQL
	Benzo(g,h,i)perylene	78	J	< PQL

BUTYL TINS ANALYSES - PSEP (Krone, et al.); GC/MS [full scan].

Butyl tin analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOP is equivalent to and referenced as PSEP (Puget Sound Estuarine Protocols; Krone, et al. 1989) with full-scan GC/MS [ion trap MS] for analysis of butyl tin compounds (monobutyltin, dibutyltin, tributyltin and tetrabutyltin).

Five catch basin sediments and three sediment pore waters were analyzed for butyl tins, which includes six primary sample locations and two blind duplicates. Sample results are presented with associated data qualifiers in Table 4-17.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and generally complete, with the exception of the porewater preparation benchsheets, surrogate spectra, and derivative formation documentation (derivative types are not identified [inspection of mass spectra indicate that hexyl derivatives were employed]). Maximum holding times are specified as 14 /40 days (sample / extract maximum holding times) for solids and 7 / 40 days for porewaters at 4 °C. (±2 °C.). Upon receipt at the laboratory, transport coolers temperatures ranged from 4 to 6.2 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

GC/MS Tuning: GC/MS tune performance was checked with 2.5 ng decafluorotriphenylphosphine (DFTPP) prior to all initial calibrations and all subsequent sample analytical runs. All sample analyses were performed within 12 hours of DFTPP checks. All ion abundances and relative ion abundances meet acceptance criteria. Review of mass spectral plots and associated mass listings supplied with the raw data show no inconsistencies or errors.

Initial Calibration: Initial multipoint calibration was performed at 1.0, 5.0, 10, 50, 100 and 200 ng/mL [on 5/16/01] for tetrabutyltin and at lower concentrations for the remaining analytes [80%, 77%, 58% and 41% for tripentyltin (surrogate), tributyltin, dibutyltin and monobutyltin, respectively]. Average Relative Response Factors (Average RRFs) ranged from 0.815 to 2.142, and %RSDs ranged from 5.9% to 18.4%. No performance criteria are available, however performance is considered reasonable and acceptable. No data was qualified based on calibration performance.

Continuing Calibration: Continuing calibrations were performed for all target analytes at the fifth calibration level (100 ng/mL tetrabutyltin and less for the other target analytes [see relative concentrations above]) prior to and following sample extract analyses. RRF %Diff's ranged from 0.5% to 18%. Performance is considered reasonable and acceptable.

Blanks: Three analytical method blanks were analyzed, two for solids and one for water matrices, as required. Method blanks showed no detections of target analytes above reporting limits.

Surrogate Compound Performance: A surrogate compound, tripentyl tin, was added to each sample (@ 1.1 µg) prior to extraction to assess analytical performance on each sample. Surrogate compound recoveries ranged from 71% to 137% in sediments and 55% to 75% in porewaters. Surrogate recovery performance is considered reasonable and acceptable.

Matrix Spike/Matrix Spike Duplicate Analyses: Matrix spike and matrix spike duplicate analyses were performed on a sediment and a porewater sample. Analyte spike concentrations were 0.178 - 0.286 µg/L for water and 114 - 183 µg/kg for sediment. MS/MSD recovery performances (as %R) are summarized as:

	Water		Sediment	
Analyte	**Recovery**	**RPD**	**Recovery**	**RPD**
tetrabutyltin	69 / 61	13	103 / 107	3.8
tributyltin	80 / 79	1.6	90 / 93	3.2
dibutyltin	40 / 41	1.2	74 / 79	6.4
monobutyltin	27 / 23	16	51 / 55	8.1

MS/MSD recoveries are typical for these types of analyses and are considered reasonable and acceptable. (Note that lower alkylated analogs exhibit lower recoveries, which is normal). No results were qualified based on MS/MSD performance.

Laboratory Control Samples: Two sets of spiked blanks (LCS) were analyzed, a set each for the solids and waters. Spike concentrations were at an equivalent of 0.12 - 0.2 µg/L and 83 - 133 µg/kg. Recoveries ranged from 29% to 92% in water and 73% to 115% in solids (TBT ranged from 67% to 88% for both matrices). Recoveries are determined to be within reasonable and acceptable ranges. LCS performances indicate the analytical systems are in control.

Target Analyte(s) Identification: Relative retention times (RRTs) for target compounds are within the CLP-specified acceptance limits (±0.06 RRT units or $\pm$ 6 seconds). Mass spectra show generally good comparability with library reference spectra. Some target analyte detections at low concentrations in water, for example dibutyltin in 010503SBDS18SS porewater, show marginal acceptance for mass spectral match and show an approximate S/N of 2. This indicates a practical lower quantitation limit of approximately 0.08 µg/L in water, and not 0.0073 and 0.0098, as indicated. Consequently, lower reporting limits have been adjusted upward due to inability to confidently identify target analytes at the lower reporting limits indicated.

Compound Quantitation and Reported Detection / Quantitation Limits: The laboratory reporting limits are lower than what can be verified from the data, particularly for waters. S/N is generally high enough to preclude identification and quantitation at the detection levels reported by the lab, especially for waters.

Lower reporting limits have been adjusted by the reviewer to concentrations that are estimated to demonstrate a S/N of 2-3 for detected analytes - this requires an upward adjustment of reporting limits by a factor of 4-5x (for example, 0.0052 µg/L has been adjusted to 0.02 µg/L [with one significant digit]). The lower reporting limit for tetrabutyltin in 010504SBDS24SS appears to not consider a dilution factor of

10x - thus the nondetect value has been adjusted from 3.4 µg/kg to 30 µg/kg [one significant digit]. Lower reporting limits should be considered estimates and have been adjusted by the reviewer to one significant digit (reported 1.7 has been adjusted to 2).

System Performance: System performance is considered generally acceptable. Major performance indicators are within acceptable limits. Lower quantitation limits (detection limits) are theoretical limits based on the absence of background and noise. Lower reporting limits have been adjusted upward to levels with a S/N of 2-3x.

No reference laboratory analyses of split samples were performed for comparison of results.

Field Replicates: Blind field splits for a sediment pair and a porewater pair were submitted and analyzed for determination of analytical variability. Sample results for replicate pairs are presented in the attached results table. Both pairs showed nondetects for all target analytes.

Overall Assessment: Most deliverables required by the project are present and data packages are generally complete, with the exception of missing documentation describing derivative formation (reaction and conditions), porewater preparation benchsheets, and some analyte spectra (surrogates and internal standards). Recommended sample holding times and conditions were met. GC/MS tuning requirements were met. Initial calibration requirements were met. Method blanks showed no detections. Reported quantitation or lower reporting limits were adjusted upward in many cases to verifiable levels. The data as reported with associated qualifiers (following adjustments made by the reviewer) are adequate for project use.

A summary of qualified results is as follows:

Sample	Analyte	Value	Qualifier	Deviation
010503SBDS18SS *pw*	Tributyl tin	0.04	U	PQL adjustment
	Tetrabutyl tin	0.04	U	PQL adjustment
010503SBDS19SS *pw*	All butyl tins	0.02	U	PQL adjustment
010503SBDS19SS *sed.*	Tetrabutyl tin	3	U	PQL adjustment
010503SBDS20SS *pw*	All butyl tins	0.02	U	PQL adjustment
010504SBDS24SS *sed.*	Tetrabutyl tin	30	U	PQL adjustment

CHLOROPHENOXY HERBICIDES ANALYSES - U.S. EPA SW-846, Method 8151A; GC/MS [full scan].

Herbicides analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOP is equivalent to and referenced as SW-846 Method 8151A with full-scan GC/MS [ion trap MS] (applying some criteria from SW-846 Method 8270) for the analysis of chlorophenoxyherbicides, 4-nitrophenol and pentachlorophenol (PCP).

Five river sediments, which includes four primary sample locations and one blind duplicate. Sample results are presented with associated data qualifiers in Table 4-5.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and generally complete, with the exception of surrogate spectra and derivative formation documentation (derivative types are not identified [inspection of mass spectra indicate that methyl ester and ether derivatives were employed]). Maximum holding times are specified as 14 /40 days (sample / extract maximum holding times) for solids at 4 °C. (±2 °C.). Upon receipt at the laboratory, transport coolers temperatures ranged from 4 to 6.2 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

GC/MS Tuning: GC/MS tune performance was checked with 2.5 ng decafluorotriphenylphosphine (DFTPP) prior to all initial calibrations and all subsequent sample analytical runs. All sample analyses were performed within 12 hours of DFTPP checks. All ion abundances and relative ion abundances meet acceptance criteria. Review of mass spectral plots and associated mass listings supplied with the raw data show no inconsistencies or errors.

Initial Calibration: Initial multipoint calibration was performed at 0.01, 0.05, 0.1, 0.5, 1.0 and 2.0 µg/mL [on 4/9/01] for target analytes and surrogate (2,4-dichlorophenylacetic acid). Average Relative Response Factors (Average RRFs) ranged from 0.374 to 1.697, and %RSDs ranged from 3.4% to 11.2%. No performance criteria are available, however performance is considered reasonable and acceptable. No data was qualified based on calibration performance.

Continuing Calibration: Continuing calibrations were performed for all target analytes at 1.0 µg/mL prior to and following sample extract analyses. RRF %Diff's ranged from 0.1% to 18%. Performance is considered reasonable and acceptable.

Blanks: One analytical method blank was analyzed, as required. Method blank results showed no detections of target analytes above reporting limits.

Surrogate Compound Performance: A surrogate compound, 2,4-dichlorophenylacetic acid, was added to each sample (@ 10 µg) prior to extraction to assess analytical performance on each sample. Surrogate

compound recoveries ranged from 65% to 93% in sediments. Surrogate recovery performance is considered reasonable and acceptable.

Matrix Spike/Matrix Spike Duplicate Analyses: Matrix spike and matrix spike duplicate analyses were performed on one sediment sample. Analyte spike concentrations were 810 µg/kg. MS/MSD recovery performances (as %R) are summarized as:

Analyte	Recoveries	RPD
dalapon	35 / 23	44
dicamba	99 / 68	37
2,4-D	96 / 86	11
pentachlorophenol	103 / 90	13
2,4,5-TP (silvex)	88 / 81	7
dinoseb	80 / 77	3
MCPP	102 / 96	7

MS/MSD recoveries are considered reasonable and acceptable. Dalapon [low] recoveries can be attributed to higher vapor pressures of the analyte and losses from concentration steps. No results were qualified based on MS/MSD performance, since no analytes were detected in project samples.

Laboratory Control Samples: A spiked blank (LCS) was analyzed at a spike level equivalent to 670 µg/kg for the following target analytes:

dalapon	35%	dicamba	88%
2,4-D	88%	pentachlorophenol	92%
2,4,5-TP	90%	dinoseb	77%
MCPP	86%		

Dalapon exhibited the lowest recovery, consistent with MS/MSD performance. Recoveries are determined to be within reasonable and acceptable ranges. LCS performances indicate the analytical systems are in control.

Target Analyte(s) Identification: No target analytes are detected or reported in project sediments.

Compound Quantitation and Reported Detection / Quantitation Limits: Reported quantitation or lower reporting limits are based on the lowest verifiable calibration point and absence of any potential interferences and baseline noise. These lower limits may not be verifiable if ionization times are reduced due to background total ionization currents and the potential use of the autogain function. Lower quantitation limits should be considered estimates.

System Performance: System performance is considered generally acceptable. Major performance indicators are within acceptable limits. Lower quantitation limits (detection limits) are theoretical limits based on the absence of background and noise.

Field Replicates: A blind field split was submitted and analyzed for determination of analytical variability. Sample results are presented in the attached results table. Both samples showed nondetects for all target analytes.

Overall Assessment: Most deliverables required by the project are present and the data package is generally complete, with the exception of missing documentation describing derivative formation (reaction and conditions), and some analyte spectra (surrogates and internal standards). Recommended sample holding times and conditions were met. GC/MS tuning requirements were met. Initial calibration requirements were met. Method blanks showed no detections. Reported quantitation or lower reporting limits should be considered estimates. The data as reported are acceptable for project use.

CHLORINATED PESTICIDES/PCBs ANALYSES in SEDIMENTS -
U.S. EPA SW-846, Methods 8081 & 8082.

Chlorinated pesticides and PCBs (Aroclors) analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOP is equivalent to and referenced as EPA SW-846 Method 8081 for analysis of chlorinated pesticides and Method 8082 for analysis of PCBs (as Aroclors) by GC/ECD. Supplied documentation shows no evidence of extract cleanups prior to instrumental analyses.

Nine river and five catch basin sediments, which includes seven primary sample locations and two blind duplicates. Sample results are presented with associated data qualifiers in Table 4-2.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and generally complete. Maximum holding times are specified as 14 /40 days (sample / extract maximum holding times) for solids at 4 °C. (±2 °C.). Upon receipt at the laboratory, transport coolers temperatures ranged from 4 to 6.2 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

GC/ECD Instrument Performance Check: DDT retention times showed less than 1% difference from a mean retention time of approximately 10.1 minutes on the primary column and approximately 10.6 minutes on the secondary or confirmatory column. DDT and endrin breakdown checks were performed every tenth run and prior to and following sample extract analyses. DDT and endrin breakdowns were less than 12% (specification is <20%) and averaged 4.2%. The integrity of the analytical system is within specification.

Initial Calibration: Six-point calibrations (1, 5, 10, 25, 50 & 75 ng/mL) were performed for pesticides [some analytes were calibrated at concentrations of x2 and methoxychlor at x10 of the above] (4/9/01) and five-point (0.01, 0.05, 0.1, 0.25 & 0.5 µg/mL) (5/15/01) for Aroclors 1221, 1242, 1254 and 1260 on a primary and confirmation (secondary) column. Three (A1221) and five (A1242, 1254 & 1260) target peaks were applied for identification and quantitation for each Aroclor. Evidence of calibration for toxaphene, Aroclors 1016, 1232 & 1248 was not included in the data package, however this may be unnecessary since these analytes were not observed in project samples. Linearity checks demonstrated individual analyte %RSDs to be within specification (specification $\leq$20%, with no more than two analytes exhibiting >30%) for pesticides. The mean RSD for the four Aroclors calibrated ranged from 9% to 22% for both columns. Initial calibrations were within acceptable limits.

Continuing Calibration or Calibration Verification: Individual pesticides mix (25 ng/mL) and Aroclors 1242 and 1260 calibration (0.1 µg/mL) checks were analyzed prior to and following every ten instrumental runs (within a 12-hour period). All analyte retention times were within the initial calibration retention time windows established above ($\pm$2 seconds on either side of the mean determined during initial calibrations). Continuing calibration responses were within the 25 RPD specification.

Blanks: Two method blanks were analyzed, one for each analytical group of 20 samples or less. No analytes were detected above the lower reporting limit.

No equipment rinsate blanks were submitted nor analyzed.

Surrogate Compound Performance: Surrogate compounds, tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCBP), were added to each sample prior to analysis to assess analytical performance. Surrogate compound recovery specifications (lab-derived control limits) are:

	Recovery acceptance ranges (%)	
Surrogate	**M.8081 (pesticides)**	**M.8082 (PCBs)**
TCMX	34 - 143	52 - 131
DCBP	26 - 157	53 - 126

All surrogate recoveries are within the above specifications. No qualification of results are required for the reported data due to surrogate performance.

Matrix Spikes/Matrix Spike Duplicates: Matrix spike and matrix spike duplicate analyses were performed on two sediment samples. Analyte spike concentrations ranged from 26 µg/kg to 52 µg/kg for pesticides and 105 µg/kg to 240 µg/kg for Aroclor 1260. Pesticide MS/MSD recoveries were evaluated against the specifications in the U.S. EPA CLP Statement of Work OLM01.0-.8 (8/91). MS/MSD recovery specifications are:

Analyte	**Recovery (%)**	**RPD**
Gamma-BHC (lindane)	46 - 127	50
Heptachlor	35 - 130	31
Aldrin	34 - 132	43
Dieldrin	31 - 134	38
Endrin	42 - 139	45
4,4'-DDT	23 - 134	50

Pesticide MS/MSD performances were within specification. Aroclor 1260 recoveries are 156 / 181% (15 RPD) and 89 / 97% (9 RPD). No data required qualification due to MS/MSD performances.

Laboratory Control Samples: Two solid spiked blanks (LCSs) were analyzed; one LCS per analytical group. All LCS recoveries were within the acceptance ranges for the MS/MSDs, above. Spike concentrations varied from an equivalent 25 - 50 µg/kg for pesticides and 100 µg/kg (based on solid concentration) for Aroclor 1260. Aroclor 1260 recovery performance was 87 - 100%. All recoveries were determined to be within acceptable range. All recovery measurements are determined to be within specification, and the analytical systems are in control.

Target Compound Identification: All reported analyte identifications and concentrations were verified on a secondary or confirmation column. Assignments were determined to be valid within a $\pm$0.003 RRT window (compared to the continuing calibration runs) on both columns, and the concentrations were determined to be within 40% (on the two columns). Some analytes exhibited elevated reporting limits due to apparent chemical interference (the concentration comparabilities on the two columns showed high

variance from coeluting interferences). This is especially the case for pesticides when elevated PCBs are present. Some pesticide lower reporting limits were elevated due to interferences from PCBs (Aroclor patterns). Aroclor patterns were examined for evaluation of accuracy in assignments - identifications appear to be appropriate. Target analyte identifications were in compliance with method specifications.

Compound Quantitation and Reported Detection / Quantitation Limits: Reported quantitation or lower reporting limits are generally based on the lowest calibration standard concentration and no chromatographic interferences. Some lower reporting limits were raised by the reviewer when PCBs interfered with the determination of pesticides. This was especially for the case of DDE, dieldrin, endrin aldehyde and heptachlor epoxide in 010502IW14SS and 010502IW15SS (factors of 2-5x). Lower reporting limits were rounded to one significant digit by the reviewer, since the lower reporting limits should be considered estimates.

Field Replicates: Blind field splits for two sediment pairs were submitted and analyzed for determination of analytical variability. Sample results for replicate pairs are presented in the attached sample results table. Results are comparable and within the variabilities typical of contaminated solids.

Overall Assessment: Quality control performance indicators were all either acceptable or within specification. Holding times and conditions are within specification. Toxaphene and some Aroclors calibrations were not found in the data deliverables. Surrogate, MS/MSD and LCS recoveries are within specification. Calibrations and endrin/DDT breakdowns are within acceptable limits. Criteria for identifications and quantitations are acceptable. Some target analyte lower reporting limits were elevated due to interferences from Aroclor constituent peaks. Data quality is sufficient for project use.

A comparison of results from split sample analyses performed by an independent reference laboratory shows nondetects for pesticides by both labs for sediment sample 010503IW14SS. A comparison of Aroclors' results for the same sample (µg/kg, dry) is as follows:

Analyte	Project Lab	Reference Lab
Aroclor 1016	10 U	230 U
Aroclor 1221	21 U	230 U
Aroclor 1232	10 U	230 U
Aroclor 1242	10 U	230 U
Aroclor 1248	10 U	230 U
Aroclor 1254	**510**	**3970**
Aroclor 1260	10 U	230 U

A comparison of PCBs results for water sample 010503IW11WCS shows nondetects for both labs.

A summary of qualified results is as follows:

Sample	Analyte	Value	Qualifier	Deviation
010502IW01SS	DDT	1.7	J	< PQL
010503IW14SS	Dieldrin	10	U	Elevated background
	Endrin	4	U	Elevated background
	Endrin aldehyde	4	U	Elevated background
	Heptachlor epoxide	4	U	Elevated background

010503IW15SS	DDE	6	U	Elevated background
	Dieldrin	9	U	Elevated background
	Endrin	3	U	Elevated background
	Endrin aldehyde	5	U	Elevated background
	Heptachlor epoxide	3	U	Elevated background
010503IW16SS	DDD	0.8	J	< PQL
	DDE	0.5	J	< PQL
010504SBDS24SS	Aroclor 1260	18	J	< PQL

PCBs (as Aroclors) ANALYSES in SEDIMENT, TISSUE and DISSOLVED and PARTICULATE WATER and SEMIPERMEABLE MEMBRANE DEVICES (SPMD)
- Battelle SOP MSL-O-009 / MSL-O-004.

PCBs (Aroclors) analyses were performed by Battelle/Marine Sciences Laboratory of Sequim, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOPs are referenced as Battelle SOP MSL-O-009, *Extraction and Clean-Up of Sediments and Tissues for Semivolatile Organics Following the Surrogate Internal Standard Method*, and MSL-O-004, *Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection*, which is based on EPA SW-846 Method 8081.

Twelve waters (six dissolved and six particulate phase waters), six river sediments, five clam composites, five crayfish composites, and five semipermeable membrane device ("fat bag") samples. Tissue concentrations are expressed normalized to wet weights, and sediments normalized to dry weights. Sample results are presented with associated data qualifiers in Tables 4-1, 4-9, and 4-10.

The analytical method reports results as recovery-corrected using the surrogates for correction. Internal standards are applied to determine surrogate recoveries for each sample. SPMD extracts were cleaned up by GPC, the water extracts required no clean up, and the sediment and tissue extracts were cleaned up by GPC and alumina chromatography.

Tissue composites consist of the following numbers of individuals:

Clams		Crayfish	
010502IW09TS	63	010509IW26TS	10
010502IW21TS	50	010509IW27TS	17
010502IW22TS	145	010509IW28TS	6
010502IW23TS	215	010509IW29TS	3
010502IW24TS	215 (split of above)	010509IW30TS	23

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are generally well organized and complete. Maximum holding times are specified as 14 /40 days (sample / extract maximum holding times) for solids at 4 °C. (±2 °C.) or extended holding times of up to one year are acceptable at temperatures of -20 °C. Solids (sediments and tissues) were frozen upon receipt at the lab and extracted after approximately 60 days. Water holding times and conditions are specified as not to exceed 7 days (as unpreserved) at 4 °C until extraction and 40 days extract holding time. Upon receipt at the laboratory, transport coolers temperatures ranged from 1.4 to 5.8 °C. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

Initial Calibration: Six-point calibrations (20, 50, 100, 200, 1000 & 5000 ng/mL) were performed for Aroclors 1248, 1254 and 1260 on a primary (DB-5) and confirmation (secondary, DB-17) column. A five-point calibration was performed for Aroclor 1242 (concentrations same as above minus the highest concentration [5000]). Initial calibration data was acquired over the period of 6/15 through 6/28. Ten to

fourteen target peaks were applied for identification and quantitation for each Aroclor on both GC columns. Quadratic fits were performed for each of the target peaks with $R^2 > 0.995$. Calibrations were established just prior to sample extract analyses. Initial calibrations were within acceptable limits.

Continuing Calibration Verification: Aroclor calibration checks (for the four mixtures identified above) were performed at 100 ng/mL just prior to and following the analysis of extracts of SPMDs and crayfish in July. RPDs were less than 30% for the analysis of crayfish and SPMD sample extracts; all within specification.

Blanks: Method blanks were analyzed for each analytical group and matrix type. No analytes were detected above the lower reporting limit.

Surrogate Compound Performance: Surrogate compounds, PCB congeners (BZ#) 103 and 198, were added to each sample (100 - 200 ng) prior to analysis to assess analytical performance and for recovery correction of reported Aroclors. Surrogate compound recovery specifications (lab-derived control limits) are 40 - 120%.

All surrogate recoveries are within the above specifications, with the exception of 010502IW03SS and 010509IW28TS which are greater than 120% (160-190%R). Sample results for 010502IW03SS and 010509IW28TS are considered estimates ("J" qualified) due to out-of-range surrogate recoveries. No other results required qualification due to surrogates performance.

Matrix Spikes/Matrix Spike Duplicates: Matrix spike and matrix spike duplicate analyses were performed on all matrices with the exception of SPMDs. Analyte spike concentrations ranged from 67 µg/kg to 100 µg/kg for Aroclor 1254 in the solid samples and 490 ng/L to 1000 ng/L in waters. The sediment and clam tissue spikes were not able to be evaluated due to native concentrations exceeding the spike levels. The crayfish spike showed a recovery of 120%, and the two water samples showed recoveries of 100% and 107%. MS/MSD recoveries are acceptable. No data required qualification due to MS/MSD performances.

Laboratory Control Samples: Spiked blanks (LCSs) were analyzed for each matrix type. Aroclor 1254 was spiked at the following levels: sediments = 67 µg/kg, tissues = 100 µg/kg, waters = 500 ng/L, and SPMDs = 1000 ng total. All LCS recoveries were within the range of 86% to 140%. All recoveries were determined to be within acceptable range. All recovery measurements are determined to be within specification, and the analytical systems are in control.

Target Compound Identification: All reported analyte identifications and concentrations were verified on the secondary or confirmation column. Concentrations were compared from the two columns and generally determined to be within 40% (on the two columns). Aroclor 1254 was always the PCB identified in all of the matrices and samples analyzed. Aroclor patterns were examined for evaluation of accuracy in assignments - identifications appear to be appropriate. Target analyte identifications were in compliance with method specifications.

Compound Quantitation and Reported Detection / Quantitation Limits: Reported quantitation or lower reporting limits are generally based on the lowest calibration standard concentration and no chromatographic interferences. Some lower reporting limits were raised by the reviewer when there was an apparent deviation from the use of the lowest calibration standard as the lower reporting limit.

Field Replicates: Blind field splits for two tissue samples, a water, a water plus particulate matter, a sediment, and an SPMD pair were submitted and analyzed for determination of monitoring variability. Sample results for replicate pairs are presented in the attached sample results table. Results are generally comparable and within the variabilities typical of contaminated materials. The water split showed especially good agreement, where the water plus particulates exhibited less agreement, as expected. Tissue and sediment pairs exhibited generally good agreement.

Overall Assessment: Quality control performance indicators were all either acceptable or within specification. Holding times and conditions are within specification. Surrogate, MS/MSD and LCS recoveries are within specification, with minor exceptions. Initial calibrations and calibration verifications are within acceptable limits. Criteria for identifications and quantitations are acceptable. Some target analyte lower reporting limits were elevated due to apparent deviations from protocol. Data quality is sufficient for project use.

A comparison of results from a split sample analysis (on 010503IW22TS, a clam homogenate) performed by an independent reference laboratory is as follows (µg/kg, wet):

Analyte	Project Lab	Reference Lab
Aroclor 1016	-	8 U
Aroclor 1221	-	8 U
Aroclor 1232	-	8 U
Aroclor 1242	14 U	8 U
Aroclor 1248	14 U	8 U
Aroclor 1254	**344**	**1522**
Aroclor 1260	14 U	8 U

A summary of qualified results is as follows:

Sample	Analyte	Value	Qualifier	Deviation
010502IW03SS *sed.*	Aroclor 1254	24000	J	Surrogate R > 120%
010502IW04WCS *water*	Aroclor 1254	0.038	J	~ PQL
010502IW05WCS *part.*	Aroclor 1254	0.032	U	Corrected PQL
010502IW06WCS *water*	Aroclor 1254	0.032	U	Corrected PQL
010503IW10WCS *water*	Aroclor 1254	0.031	U	Corrected PQL
010503IW10WCS *part.*	Aroclor 1254	0.032	U	Corrected PQL
010503IW11WCS *water*	Aroclor 1254	0.030	U	Corrected PQL
010509IW28TS *crayfish*	Aroclor 1254	75600	J	Surrogate R > 120%

METALS ANALYSES - U.S. EPA SW-846, Methods 6010B, 6020 & 7471A.

Metals analyses were performed by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and referenced SOPs. The analytical SOPs are equivalent to and referenced as EPA SW-846 Method 6010B for analysis of barium (Ba), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), vanadium (V) and zinc (Zn); Method 6020 for analysis of antimony (Sb), arsenic (As), beryllium (Be), cadmium (Cd), cobalt (Co), selenium (Se), silver (Ag) and thallium (Tl); and Method 7471A for the determination of mercury (Hg).

Five river and five catch basin sediments, which include eight primary sample locations and two blind duplicates. Sample results are presented with associated data qualifiers in Table 4-6 and 4-15.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are well organized and generally complete. Maximum holding times for solids are specified as 28 days for mercury and 6 months for other metals. Holding conditions and times are determined to be acceptable. No results require qualification due to holding times and conditions.

Initial Calibration: The laboratory performed initial instrumental calibrations daily using at least the minimum required number of data points to establish the analytical curve for each method: a blank and one standard for ICP-AES analyses, a blank and five standards for ICP-MS analyses, and a blank and five standards for mercury analyses. Correlation coefficients for all mercury initial calibrations are $\geq$ 0.995, as required.

Initial Calibration Verification: The laboratory performed initial calibration verification checks (ICVs) immediately after initial instrumental calibrations during all ICP and mercury analytical sequences, as required. All ICV recoveries are within acceptance limits (90–110% for ICP; 80–120% for mercury).

Continuing Calibration Verification: The laboratory analyzed continuing calibration verification standards (CCVs) at the required frequency for all ICP and mercury analytical sequences (at the beginning and end of each run; at a frequency of = 10% or every two hours, whichever is more frequent). All CCV recoveries are within acceptance limits (90–110% for ICP; 80–120% for mercury).

Blanks: Initial calibration blanks (ICBs) were analyzed immediately after ICVs, and continuing calibration blanks (CCBs) were analyzed immediately after CCVs during all ICP and mercury analytical sequences, as required.

Two preparation or method blanks were analyzed for all target analytes. Sb, Pb, Mn and Ni were detected at levels less than the PQLs. All associated sample results are qualified as nondetected at the associated values ("U" qualifier code).

No field rinsate blanks were submitted for analysis.

Interference Check Samples: ICP interference check solutions (ICS) were analyzed for the target analytes at the beginning of each ICP analytical run, as required by the individual methods. Recoveries for all required target analytes in all check samples are within acceptance limits (80–120%).

Laboratory Control Samples: Laboratory control samples (LCS) were analyzed at the required frequency (at least one sample per matrix per preparation batch). All LCS results are within 80–120% of known values.

Duplicate Sample Analyses: Laboratory duplicate samples were analyzed for the target analytes at the required frequency (at least one sample per matrix per preparation batch). Acceptance limits applied in this evaluation of duplicate sample analyses are as: Results = 5X the reporting limit, = 35% Relative Percent Difference (RPD) for solids; results < 5X the reporting limit ± 2X the reporting limit). Results of all duplicate analyses meet these criteria with the exception of manganese in one duplicate analysis of a sediment sample (010501SBMS01SS) (the other two duplicate pairs showed acceptable performance). Manganese in the associated sample only is qualified as estimated ("J").

Matrix Spike Sample Analyses: Matrix spike samples were analyzed for the target analytes in three samples. Acceptance limits for matrix spike recovery are 75–125% and are applicable only to those samples and analytes for which the sample concentration does not exceed four times the spike concentration. Some recoveries were outside the acceptance range due to high native concentrations relative to spike levels. No results required qualification based on matrix spike performances.

Reported Detection/Quantitation Limits: Reported quantitation or lower reporting limits are within reasonable ranges and allow comparison to background and/or reference levels.

Field Replicates: Two blind field replicate sample pairs were submitted and analyzed for metals for determination of analytical variability. Sample results for replicate pairs are presented in the attached table. Greatest variability is associated with Pb and Ni in catch basin sediments; 90% and 95%, respectively. Comparability (or lack of) is not atypical of contaminated solids. No results are qualified due to blind duplicate analytical performance.

Overall Assessment: All deliverables required by the project are present and data packages are generally complete. All analyses meet recommended sample holding times. Initial calibration and calibration verification are acceptable. Sb, Pb, Mn and Ni were detected in method blanks at levels less than the PQL; affected results were qualified as nondetected at the associated values. Recoveries for interference check samples and laboratory control samples are acceptable. Laboratory duplicate sample analyses are acceptable with the exception of manganese (Mn) in one sediment analysis; associated positive results are qualified as estimated ("J"). Matrix spike recovery performances are within acceptable ranges. Reported quantitation or lower reporting limits are sufficient for comparison to background and/or proposed screening levels. Overall analytical performance is considered acceptable and the data quality is sufficient for project use.

Comparison of split sample analyses performed by the project lab and an independent reference lab is as follows (mg/kg, dry):

	010501SBMS01SS		010503IW14SS	
Analyte	**Project lab**	**Ref. lab**	**Project lab**	**Ref. lab**
Arsenic	3.6	4.4	2.4	3.6
Silver	0.26	1.0 U	0.16	1.0 U
Aluminum	9500	4810	11000	8210
Barium	76	45.1	88	88.8
Beryllium	0.14	0.17 J	0.27	0.45
Calcium	4000	3030	4200	4950
Cadmium	1.6	0.90	1.0	0.36 J
Cobalt	11	15.4	8.6	11.1
Chromium	87	540	16	19.8
Copper	43	45.1	27	23.6
Iron	28000	27900	18000	20400
Potassium	450 J	280	1100	739
Magnesium	6000	17400	4600	5530
Manganese	510 J	349	380	305
Sodium	-	300	-	400
Nickel	33	263	7.6	16.4
Lead	630	280	9.3	8.6
Selenium	0.72 U	4.0 U	0.7 U	4.0 U
Antimony	1.2	1.9 J	0.70 U	1.7 J
Thallium	0.032 J	6.0 U	0.16	6.0 U
Vanadium	50	42.3	44	54.8
Zinc	180	174	73	96.5
Mercury	0.063	0.016	0.035	0.028

A summary of qualified results is as follows:

Sample	Analyte	Value	Qualifier	Deviation
010501SBMS01SS	Manganese	510	J	High duplicate variability
	Potassium	450	J	< PQL
	Thallium	0.032	J	< PQL
010501SBMS02SS	Potassium	300	J	< PQL
	Beryllium	0.11	J	< PQL
	Selenium	0.39	J	< PQL
	Silver	0.11	J	< PQL
	Thallium	0.047	J	< PQL
010502IW01SS	Antimony	0.62	U	Blank contamination
	Selenium	0.26	J	< PQL
010502IW07SS	Antimony	0.72	U	Blank contamination
	Selenium	0.31	J	< PQL
	Silver	0.11	J	< PQL
010503IW14SS	Antimony	0.70	U	Blank contamination

010503IW15SS	Antimony	0.73	U	Blank contamination
010503IW16SS	Antimony	0.70	U	Blank contamination
	Mercury	0..017	J	< PQL
	Selenium	0.2	J	< PQL
010503SBDS18SS	Antimony	0.92	U	Blank contamination
010503SBDS19SS	Antimony	0.97	U	Blank contamination
	Beryllium	0.086	J	< PQL
	Mercury	0.021	J	< PQL
	Potassium	340	J	< PQL
	Selenium	0.19	J	< PQL
	Silver	0.099	J	< PQL
	Thallium	0.072	J	< PQL
010504SBDS24SS	Antimony	1.5	U	Blank contamination
	Mercury	0.034	J	< PQL

TOTAL ORGANIC CARBON (TOC) ANALYSES - U.S. EPA SW-846, Method 9060 & ASTM D4129-82M.

TOC analyses were performed on fourteen sediments by Sound Analytical Services, Inc. (SAS) of Tacoma, Washington, in accordance with the requirements of the *Sampling and Analysis Plan, In-Water Investigation, Bradford Island Landfill,* April 2001 (URS) and SW-846 Method 9060. Analyses of six river sediments were analyzed for TOC by Columbia Analytical Services, Inc. (CAS) of Kelso, Washington, by ASTM Method D4129-82 (modified for solids). Sample results are presented with associated data qualifiers in Table 4-1 and 4-3.

Sample Documentation, Custody and Holding Conditions / Times: All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are generally well organized and complete. Maximum holding times for solids are specified as 28 days at 4 °C. Holding conditions and times are determined to be acceptable for samples handled and analyzed by SAS. Holding times for samples analyzed by CAS are determined to be approximately 16 weeks. Holding conditions are reported to be as frozen samples while in the custody of Battelle Marine Sciences Laboratory for 15 weeks (regional guidance recommends a maximum holding time of one year at -20 °C). Results for samples handled by both SAS and CAS/Battelle require no qualification due to holding times and conditions. No results require qualification due to sample holding times and conditions.

Calibration: SAS performed initial instrumental calibration with a 1.0% standard and followed sample analyses with (what appears to be) verification standards at 0.5%, 0.2%, 5.0% and 6.0%. An NIST check sample showed a 93% and 95% recovery at a concentration of 3.35%. CAS did not document an initial calibration, however continuing calibration verifications were documented twice showing 96% and 98% recoveries at a concentration of 20.0%.

Blanks: Initial calibration blanks (ICBs) and continuing calibration blanks (CCBs) were analyzed and demonstrated no detections.

Method blanks were analyzed and reported by both labs. No detections were found above lower reporting limits.

Laboratory Control Samples: Laboratory control sample (LCS) results were submitted by CAS. LCS performance was 103% of the known value. SAS ran an NIST2704 check sample for each batch and reported a 93% and 95% recovery.

Duplicate Sample Analyses: A laboratory duplicate analysis was performed by CAS on sample 010502IW11SS. RPD was reported at 15%, well within the specification of = 35% Relative Percent Difference (RPD) for solids.

Matrix Spike Sample Analyses: One matrix spike analysis was performed by CAS on sample 010502IW11SS, which reported a recovery of 87%. Acceptance limits for matrix spike recovery are 75–125%. SAS performed three sets of MS/MSD analyses for TOC. Recoveries ranged from 98% to

117%, with RPDs ranging from 0.8% to 19%. No results required qualification based on matrix spike performances.

Field Replicates: Three blind field replicate sample pairs were submitted and analyzed for determination of monitoring variability. Sample results for replicate pairs are presented in the attached table. Greatest variability is associated with river sediments; 58% Diff (or RPD). This variability is consistent with the variability observed for other contaminants (PCBs) reported in the same sample. No results are qualified due to blind duplicate analyses.

Overall Assessment: All deliverables required by the project are present and data packages are generally complete. Sample holding times and conditions are determined to be acceptable. Initial calibrations and calibration verifications are acceptable. Method blanks showed no detections above lower reporting limits. LCS and matrix spike recovery performances are within acceptable ranges. Overall analytical performance is considered acceptable and the data quality is sufficient for project use.

Comparison of split sample analyses performed by the project lab and an independent reference lab is as follows (%, dry):

	010501SBMS01SS		**010503IW14SS**	
Analyte	**Project lab**	**Ref. lab**	**Project lab**	**Ref. lab**
TOC	1.2	1.1	0.29	0.17